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Welcome

# **An Introduction to the Revised NR 149**

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WDNR Laboratory Certification  
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## **Examples from NR 149 Presentation**

The following slides are the examples from "An Introduction to  
the Revised NR 149- Parts I and II"

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## **An Intro to the Revised NR 149**

Fields of Certification and Registration  
Examples

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## Fields of Registration Small WPDES Laboratory

Current NR 149		Proposed NR 149	
		<i>Aqueous Matrix</i>	
Category	Analyte	Analytical Technique	Analyte
01- Oxygen Utilization	BOD Carbonaceous BOD	Electrometric Assays	BOD Carbonaceous BOD
02- Nitrogen	Ammonia as N	Electrometric Assays	Ammonia
03- Phosphorus	Total Phosphorus	Colorimetric or Nephelometric	Phosphorus, Total
04- Physical	TSS	Gravimetric Assays	Residue, Non-filterable

This identifies the current registrations maintained by a WPDES laboratory and the equivalent registrations under the proposal.

New certificates will look different than the current ones, but the new format will more accurately reflect the analytical capabilities of a laboratory.

For instance, we can see from the new structure that this laboratory analyzes ammonia using an ion selective electrode and phosphorus using a spectrophotometer.

## Current Scope Small WPDES Laboratory

### Category 01 – Oxygen Utilization

Biochemical Oxygen Demand  
Carbonaceous BOD

### Category 02 – Nitrogen

Ammonia as N

### Category 03 – Phosphorus

Total Phosphorus

### Category 04 – Physical

Total Suspended Solids

This current scope is sent to the laboratory as an attachment to the actual “certificate”.

This laboratory maintains registration for BOD, CBOD, Ammonia, Total Phosphorus and TSS.

## Post-Revision Scope Small WPDES Laboratory

### *Aqueous Matrix*

#### Electrometric Assays

Biochemical Oxygen Demand  
Carbonaceous BOD  
Ammonia

#### Colorimetric or Nephelometric

Phosphorus, Total

#### Gravimetric Assays

Residue, Nonfilterable

The post-revision scope identifies the matrix (aqueous), analytical techniques (electrometric assays, colorimetric or nephelometric, and gravimetric assays) and the corresponding analytes for which this laboratory maintains registration.

## Fields of Certification Small SDWA Laboratory

Current NR 149	Proposed NR 149	
Category 18- Safe Drinking Water	<i>Drinking Water Matrix</i>	
	Analytical Method	Analyte
Fluoride- EPA 300.0	EPA 300.0	Fluoride
Nitrate- SM 4500-NO <sub>3</sub> D, SM 20 <sup>th</sup> ed.	4500-NO <sub>3</sub> D, SM 20 <sup>th</sup> ed	Nitrate
Copper- SM 3111B, 18/19 <sup>th</sup> ed.	SM 3111B, 18/19 <sup>th</sup> ed.	Copper
Lead- SM 3113B, 18/19 <sup>th</sup> ed.	SM 3113B, 18/19 <sup>th</sup> ed.	Lead

For SDWA certification, the new scopes are not very different from the old ones. We are currently capturing method information for SDWA laboratories. The order of the information is reversed in the new scope.

## Current Scope Small SDWA Laboratory

### Category 18- Safe Drinking Water

Fluoride- EPA 300.0

Nitrate- SM 4500-NO<sub>3</sub> D, SM 20<sup>th</sup> ed.

Copper- SM 3111B, 18/19<sup>th</sup> ed.

Lead- SM 3113B, 18/19<sup>th</sup> ed.

## Post-Revision Scope Small SDWA Laboratory

### *Drinking Water Matrix*

EPA 300.0- Fluoride

SM 4500-NO<sub>3</sub> D, SM 20<sup>th</sup> ed.- Nitrate

SM 3111B, 18/19<sup>th</sup> ed.- Copper

SM 3113B, 18/19<sup>th</sup> ed.- Lead

The only difference between the current and post-revision scopes for certification in the drinking water matrix is the order of analytical method and analyte.

## Fields of Certification WPDES Pretreatment Program

Current NR 149		Proposed NR 149	
Category		Analyte	
04- Physical	Oil & Grease, HEM	Gravimetric	Oil & Grease as HEM
06- General II	Cyanide	Colorimetric or Nephelometric	Cyanide
08- Metals I	Cadmium Chromium, Total Copper Lead Nickel Silver Zinc	Flame AA  ICP	Copper Nickel Zinc Cadmium Chromium, Total Silver

In this example, this wastewater treatment plant laboratory opted for certification voluntarily.

This laboratory also maintains certifications for BOD, Ammonia, Total Phosphorus and Solids (TSS, TS, and TVSS) to comply with its own permit requirements (but they are not included in this example, for the sake of brevity).

The tests in this example are required as a result of a local pretreatment ordinance- the plant uses this data for billing pretreatment program participants. The ordinance requires pre-treatment analyses to be performed by a certified or registered laboratory.

## Current Scope WPDES w/ Pretreatment

### Category 04- Physical

Oil & Grease (HEM)

### Category 06- General II

Cyanide

### Category 08- Metals I

Cadmium  
Copper  
Chromium, Total  
Lead  
Nickel  
Zinc

## Post-Revision Scope WPDES w/ Pretreatment

### Aqueous Matrix

Colorimetric or Nephelometric

Cyanide

### Gravimetric Assays

Oil & Grease as HEM

### Flame AA

Copper  
Nickel  
Zinc

### ICP

Cadmium  
Chromium, Total  
Lead

This example clearly indicates the technique used for the metals determinations; this information would not be obvious in the current scope.

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## Fields of Certification Commercial Lab Pesticides

Current NR 149		Proposed NR 149	
		<i>Aqueous Matrix</i>	
Category	Analyte	Analytical Technique	Analyte
13- Liquid Chromatography	Carbamates	HPLC	Aldicarb Barban
14- Pesticides	Organophosphorus Pesticides Triazines & Metabolites	GC GC	Dimethoate Famfur Atrazine Cyanazine Simazine
16- Organics; Organochlorine	Organochlorine Pesticides	GC-MS	Organochlorine Pesticides Analyte Group
		GC	Organochlorine Pesticides Analyte Group
19- Any Single Analyte	2,4,5-T 2,4,5-TP	LC-MS	2,4,5-T 2,4,5-TP

This is an example of a commercial laboratory that analyzes some common and some unusual pesticides in the aqueous matrix.

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## Fields of Certification Commercial Lab Pesticides

Organochlorine Pesticides Analyte Group		
Aldrin	4,4'-DDE	Heptachlor Epoxide
alpha-BHC	4,4'-DDT	Isodrin
beta-BHC	Dichloran	Kepone
delta-BHC	Dieldrin	Methoxychlor
gamma-BHC	Endosulfan I	Mirex
Captafol	Endosulfan II	PCNB
Captan	Endosulfan Sulfate	Perthane
Chlordane	Endrin	Strobane
Chloroneb	Endrin Aldehyde	Toxaphene
4,4'-DDD	Heptachlor	

The organochlorine pesticides analyte group includes all the pesticides listed on this slide.

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## Fields of Certification Commercial Lab Pesticides

Current NR 149		Proposed NR 149	
		<i>Solids Matrix</i>	
Category	Analyte	Analytical Technique	Analyte
14- Pesticides	Organophosphorus Pesticides Triazines & Metabolites	GC	Chlordane Toxaphene
16- Organics; Organochlorine	Organochlorine Pesticides	GC-MS	Organochlorine Pesticides Analyte Group Dimethoate Famfur Atrazine Cyanazine Simazine

This example commercial laboratory also analyzes some of the common and unusual pesticides in solids, as well.

## Current Scope Commercial Lab Pesticides

### Category 13- Liquid Chromatography

Carbamates

### Category 14- Pesticides

Organophosphorus

Triazines and Metabolites

### Category 16- Organics; Organochlorines

Organochlorine Pesticides

## Post-Revision Scope Commercial Lab Pesticides

### *Aqueous Matrix*

GC-MS

Organochlorine Pesticides  
Analyte Group

GC

Organochlorine Pesticides  
Analyte Group  
Dimethoate  
Famfur  
Atrazine  
Cyanazine  
Simazine

HPLC

Aldicarb  
Barban

LC-MS

2,4,5-T  
2,4,5-TP

### *Solids Matrix*

GC

Chordane  
Toxaphene

GC-MS

Organochlorine Pesticides  
Analyte Group  
Dimethoate  
Famfur  
Atrazine  
Cyanazine  
Simazine

The new scope indicates the techniques used to analyze each pesticides under certification. It also allows laboratories to tailor their scopes to the analytes requested for individual matrices. Note the differences between the analytes and techniques for the aqueous and solids matrices.

## An Intro to the Revised NR 149

Fee Examples

## Fees Small WPDES Laboratory

Current Fees	RVU
Registration Base Fee	10
Category 01- Oxygen Utilization	1
Category 02- Nitrogen	1
Category 03- Phosphorus	1
Category 04- Physical	1
<b>Total:</b>	<b>14</b>
Proposed Fees	RVU
Base Fee, Registration	5
Matrix Fee, Aqueous	5
Technology Fees:	
Colorimetric or Nephelometric	2
Electrometric Assays	1
Gravimetric Assays	1
<b>Total:</b>	<b>14</b>

Note that the RVUs for a typical small wastewater treatment plant laboratory are not increasing as a result of this proposal.

## Fees Small SDWA Laboratory

Current Fees	RVU
Certification Base Fee	15
Category 18- Safe Drinking Water	20
<b>Total:</b>	<b>35</b>
Proposed Fees	RVU
Base Fee, Certification	10
Matrix Fee, Drinking Water	5
Analytical Class Fees:	
Copper and Lead	4
Nitrate, Nitrite, Nitrate + Nitrite and Fluoride	2
<b>Total:</b>	<b>21</b>

Note that this small SDWA laboratory will experience a drop in assessed RVUs. This laboratory is exempted from paying the minimum certification fee. The assessed 21 RVUs are lower than the minimum certification fee of 24 RVUs.

## Fees WPDES w/ Pretreatment

Current Fees	RVU
Certification Base Fee	15
Category 04- Physical	1
Category 06- General II	2
Category 08- Metals	4
<b>Total:</b>	<b>22</b>
Proposed Fees	RVU
Base Fee, Certification	10
Matrix Fee, Aqueous	5
Technology Fees, Aqueous Matrix:	
Colorimetric or Nephelometric Assays	2
Gravimetric Assays, Oil & Grease	1
Flame Atomic Absorption Spectrophotometry	3
ICP	3
<b>Total</b>	<b>24</b>

This is the same wastewater treatment laboratory that voluntarily opted for certification.

In this case, the laboratory will see a slight increase of 2 RVUs under the proposed code. Should the laboratory decide to become registered, the RVUs assessed to the laboratory would decrease by 5 for a total of 19 RVUs.

**Fees**  
**Commercial Pesticides Only**

Current Fees	RVU
Certification Base Fee	15
Category 13- Liquid Chromatography	4
Category 14- Pesticides	4
Category 16- Organochlorine Compounds	4
Category 19- Any Single Analyte	4
<b>Total:</b>	<b>31</b>
Proposed Fees	RVU
Base Fee, Certification	10
Matrix Fee, Aqueous	5
Matrix Fee, Solids	5

**Fees**  
**Commercial Pesticides Only**

Proposed Fees, continued	
Technology Fees, Aqueous Matrix:	
HPLC	4
Gas Chromatography	3
Gas Chromatography-Mass Spectrometry	4
Liquid Chromatography-Mass Spectrometry	5
Technology Fees, Solids Matrix	
Gas Chromatography	3
Gas Chromatography-Mass Spectrometry	4
<b>Total:</b>	<b>43</b>

This laboratory will experience an increase in assessed RVUs.

The sum of all the RVUs for pesticide analytical classes is 16, which is right at the cap for maximum fees that can be assessed for certification or registration for pesticides. If the laboratory added another analytical techniques for pesticides, the laboratory would not be assessed any additional fees. For example, if the laboratory opted to obtain certification for immunoassay atrazine, it would pay no additional fee.

**Example Maximum Fees**  
**Multiple Techniques for Metals**

Current			Proposed		
		RVU	Aqueous Matrix		RVU
Category 08- Metals I	Arsenic	4	CVAA FLAA	Mercury	3
	Selenium			Copper	3
	Lead			Magnesium	
	Copper		GFAA	Zinc	
	Mercury			Arsenic	3
	Chromium		ICP	Selenium	
	Chromium			Chromium	3
	Magnesium		ICP-MS	Copper	
				Lead	
				Magnesium	
Category 09- Metals II		4		Arsenic	4
	Titanium			Chromium	
				Lead	
				Selenium	
				Titanium	
<b>Total</b>		<b>8</b>		<b>Total</b>	<b>16</b>

RVU exceeds maximum for metals analyte class-  
lab pays maximum 10 RVU for aqueous matrix

The illustrates how the maximum fee for the metals analytical class would save this laboratory the cost of 6 RVUs when getting registered or certified in the aqueous matrix for multiple techniques to analyze metals.



Example  
Metals Maximum Fee

Current			Proposed		
		RVU	Solids Matrix		RVU
Category 08- Metals I	Arsenic	4	CVAA	Mercury	3
	Selenium		FLAA	Copper	3
	Lead			Magnesium	
	Copper			Zinc	
	Mercury		ICP	Chromium	3
	Chromium			Copper	
	Magnesium			Lead	
			ICP-MS	Magnesium	
				Arsenic	4
				Chromium	
				Lead	
				Selenium	
				Titanium	
Category 09- Metals II	Titanium	4			
	Total	8		Total	13

RVU exceeds maximum for metals analyte class-  
lab pays maximum 10 RVU for solids matrix as well

The illustrates how the maximum analytical class fee would save this laboratory 3 RVUs when getting certified or registered in the solid matrix for multiple techniques for analyzing metals.

An Intro to the Revised NR 149

Proficiency Testing Examples

Required PTs  
Small WPDES Laboratory

Aqueous Matrix

Electrometric Assays

Biochemical Oxygen Demand	WP
Carbonaceous BOD	WP
Ammonia	WP

Colorimetric or Nephelometric

Phosphorus, Total	WP
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Gravimetric Assays

Residue, Nonfilterable	WP
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This identifies the commonly available types of PT for the listed analytical techniques. To obtain certification for CBOD laboratories will now have to analyze a PT. However, the same PT that is analyzed for BOD can be also analyzed for CBOD.

Required PTs Small SDWA Laboratory	
<b>Drinking Water Matrix</b>	
EPA 300.0- Fluoride	WS
SM 4500-NO <sub>3</sub> D, SM 20 <sup>th</sup> ed.- Nitrate	WS
SM 3111B, 18/19 <sup>th</sup> ed.- Copper	WS
SM 3113B, 18/19 <sup>th</sup> ed.- Lead	WS

PTs for the drinking water matrix are analyzed by method, and come from the WS series offered by most providers, as is currently the case.

Required PTs WPDES w/ Pretreatment	
<b>Aqueous Matrix</b>	
<b>Colorimetric or Nephelometric</b>	
Cyanide	WP
<b>Gravimetric Assays</b>	
Oil & Grease as HEM	WP
<b>Flame AA</b>	
Copper	Technique Exempt
Nickel	Technique Exempt
Zinc	Technique Exempt
<b>ICP</b>	
Cadmium	WP
Chromium, Total	WP
Lead	WP

This shows the current PT exemptions proposed for flame AA.

Required PTs Commercial Pesticides		
<b>Aqueous Matrix</b>		<b>Solids Matrix</b> <b>None</b>
GC-MS	Organochlorine Pesticides Analyte Group	WP
GC	Organochlorine Pesticides Analyte Group	WP
	Dimethoate	?
	Famfur	?
	Atrazine	WS?
	Cyanazine	WS?
	Simazine	WS?
HPLC	Aldicarb	WS?
	Barban	?
LC-MS	2,4,5-T	WS?
	2,4,5-TP	WS?
GC-MS	Organochlorine Pesticides Analyte Group	Dimethoate
GC	Famfur	Atrazine
	Cyanazine	Simazine
GC	Chlordane	Toxaphene

PTs for organochlorine pesticides are readily available, but that is not necessarily the case for herbicides, carbamates, organophosphorus pesticides and trazines.

Providers have historically re-labeled WS ampules for WP herbicides but only included four herbicides in the ampules. This illustrate the current uncertainty about the availability of PTs for some pesticides in the aqueous matrix and most pesticides in the solid matrix. This is one reason why the proposal calls for publishing a list of required PTs and approved providers annually.

## An Intro to the Revised NR 149

### Support Equipment Calibration and Verification Examples

### Support Equipment Calibration or Verification

Equipment	Method	Frequency
Thermometers, thermocouples, infrared guns	NIST-traceable thermometer	Yearly
Analytical balances	NIST-traceable weights- 1 gm range, 1 mg range	Monthly
Non-analytical balances	NIST-traceable or verifiable- range of use	Monthly
Mechanical and automatic micro-pipettes, burets, dilutors and dispensers	Verify volume transferred gravimetrically	Quarterly
Volumetric glassware and syringes, Class A		Exempted
Disposable pipettes, used in method steps		Exempted

This summarizes the method and frequency for calibrating or verifying support equipment.

Note that these procedures would not apply to analytical instruments such as spectrophotometers.

### Sample Testing and Holding Calibration or Verification

Equipment	Criteria	Frequency
Refrigerators for sample storage	Above freezing to 6° C	Daily, when in use
Thermostats to be set so that temperature is maintained on days samples are stored		
Autoclaves, incubators, ovens & water baths for sample processing	Method-specified	Daily, when in use
BOD incubator thermostats to be set so that temperature is maintained on days samples are processed		

This summarizes the criteria used for determining the calibration state of support equipment that holds samples and the frequency of the verification. The proposal does not require laboratory personnel to monitor the temperature of refrigerators and incubators on days when personnel are not scheduled to be present to perform analyses.

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## **An Intro to the Revised NR 149**

### Analytical Batch Examples

The following slides summarize requirements for calibration and quality control in the context of some typical analyses.

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### **Example #1**

Analytical Test: Automated Colorimetric Total Phenolic Compounds

Calibration routine: 3 standards, Linear

Analytical batch: 30 samples, distilled in 2 preparation batches

This example illustrates what the laboratory would do on a day when it is performing an initial calibration.

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### **Example #1 Initial Calibration**

Standard 1, source A

Standard 2, source A

Standard 3, source A

Initial Calibration Verification, source B

Method Blank 1 (from preparation batch 1)

Laboratory Control Sample 1 (preparation batch 1), source B

## Example #1 Initial Calibration

Samples 1-20

Continuing Calibration Verification standard, source A

Method Blank 2 (from preparation batch 2)

Laboratory Control Sample 2 (preparation batch 2), source B

Samples 21-30

Closing Continuing Calibration Verification standard, source A

## Example #1, Initial Calibration, in Summary

- |        |                                     |
|--------|-------------------------------------|
| 1-3.   | 3-point Calibration Curve, source A |
| 4.     | ICV, source B                       |
| 5.     | MB 1                                |
| 6.     | LCS 1 source B                      |
| 7-26.  | Samples 1-20                        |
| 27.    | CCV, source A                       |
| 28.    | MB 2                                |
| 29.    | LCS 2, source B                     |
| 30-39. | Samples 21-30                       |
| 40.    | Closing CCV, source A               |

If the laboratory analyzes QCS, the ICV can be eliminated.

If the laboratory analyzes matrix spikes and evaluates them against the acceptance criteria for Laboratory Control Samples, the LCS can be eliminated.

Analysis of MS/MSD or replicates is only required if specified by method and there is sufficient sample volume to do it.

## Example #1 No Calibration

Continuing Calibration Verification standard, source A

Method Blank 1 (from preparation batch 1)

Laboratory Control Sample 1 (preparation batch 1), source B

Samples 1-20

Continuing Calibration Verification standard, source A

This example illustrates what the laboratory would do on a day when it is not performing an initial calibration.

### Example #1 No Calibration

Method Blank 2 (from preparation batch 2)

Laboratory Control Sample 2 (preparation batch 2), source B

Samples 21-30

Closing CCV standard, source A

If the laboratory analyzes QCS, the LCS can be eliminated.

Analysis of MS/MSD or replicates is only required if specified by method and there is sufficient sample volume to do it.

### Example #1 No Calibration, in Summary

1. CCV, source A
2. MB 1
4. LCS 1, source B
- 5-24. Samples 1-20
25. CCV, source A
26. MB 2
27. LCS 2, source B
- 28-37. Samples 21-30
38. Closing CCV, source A

If the laboratory analyzes matrix spikes and evaluates them against the acceptance criteria for Laboratory Control Samples, the LCS can be eliminated.

Analysis of MS/MSD or replicates is only required if specified by method and there is sufficient sample volume to do it.

### Example #2

Analytical Test: Ion Selective Electrode Ammonia

Calibration Routine: 3 standards, Linear Curve

log [standard concentration] v. mV response

Analytical Batch: 6 samples, undistilled

Because ion selective electrodes require daily calibration, this is what the proposal requires for ammonia analysis.

## Example #2 Initial Calibration

0.2 mg/L standard, source A

2.0 mg/L standard, source A

20 mg/L standard, source A

Method Blank

Laboratory Control Sample, source B

Samples 1-6

Closing Continuing Calibration Verification standard, source A

## Example #2, Initial Calibration, in Summary

1-3. 3-point Calibration, source A

4. MB

5. LCS, source B

6-11. Samples 1-6

12. Closing CCV, source A

If the laboratory analyzes matrix spikes and evaluates them against the acceptance criteria for Laboratory Control Samples, the LCS can be eliminated.

## Example #3

Analytical Test: GC/MS BNAs

Calibration routine: 5 Standards, Quadratic

Analytical batch: 40 Samples, extracted in 2  
preparation batches

For this example, to illustrate the requirements of the code proposal, we have assumed that the laboratory is not bound to follow a method that contains more stringent calibration and quality control procedures. When methods contain more stringent calibration and quality control procedures than those specified in the code, the laboratory must follow the more stringent procedures.

### Example #3 Initial Calibration

STDs 1-5, source A

ICV, source B

Method Blank 1, (from prep batch 1)

Laboratory Control Sample 1 (prep batch 1), source B

Samples 1-20

CCV-1, source A

### Example #3 Initial Calibration

CCV- 2, source A

Method Blank 2, (from prep batch 2)

LCS 2 (from prep batch 2) , source B

Samples 21-40

Closing CCV-1 source A

Closing CCV- 2, source A

### Example #3, Initial Calibration, in summary

- |        |                               |
|--------|-------------------------------|
| 1-5.   | 5-point Calibration, source A |
| 6.     | ICV, source B                 |
| 7.     | MB 1                          |
| 8.     | LCS 1, source B               |
| 9-28.  | Samples 1-20                  |
| 29.    | CCV 1, source A               |
| 30.    | CCV 2, source A               |
| 31.    | MB 2                          |
| 32.    | LCS 2, source B               |
| 33-52. | Samples 21-40                 |
| 53.    | Closing CCV 1, source A       |
| 54.    | Closing CCV 2, source A       |



**Example #3  
No Calibration**

CCV-1, source A  
CCV-2, source A  
Method Blank 1, (from prep batch 1)  
Laboratory Control Sample 1 (from prep batch 1), source B  
Samples 1-20  
CCV-1, source A

This example illustrates what the laboratory would do on a day when it is not performing an initial calibration.

**Example #3  
No Calibration**

CCV- 2, source A  
Method Blank 2 (from prep batch 2)  
LCS 2 (from prep batch 2), source B  
Samples 21-40  
Closing CCV-1, source A  
Closing CCV-2, source A

**Example #3,  
No Calibration, in Summary**

- 1. CCV 1, source A
- 2. CCV 2, source A
- 3. MB 1
- 4. LCS 1, source B
- 5-24. Samples 1-20
- 25. CCV 1, source A
- 26. CCV 2, source A
- 27. MB
- 28. LCS 2, source B
- 29-48. Samples 21-40
- 49. Closing CCV 1, source A
- 50. Closing CCV 2, source A

**Example #4**

Analytical Test: BOD

Calibration Routine: DO Meter calibrated each day  
of use with water-saturated air

Analytical batch: 2 influents, 2 effluents plus seed  
controls

**Example #4  
Initial Calibration**

Method Blank (dilution water)

LCS (glucose-glutamic acid)

Seed control (minimum 2 dilutions)

Influents 1 and 2

Effluents 1 and 2

DO meter calibrated using water-saturated air;  
laboratory records temperature, barometric  
pressure and resulting calibration value.

**Example #4,  
Initial Calibration, in summary**

1. Standardize DO Meter
2. MB
3. LCS (GGA)
- 4-5. Seed Controls
- 6-9. Samples 1-4

**Example #5**

Analytical Test: Solids, Nonfilterable (TSS)  
Calibration Routine: Analytical balance verified  
monthly in gm- and mg-range  
Analytical batch: 1 influent, 1 effluent

**Example #5  
Balance Verification & Procedure**

Balance verified with Class I weights in gm and mg-range  
monthly  
Filter tare weights determined  
Samples filtered and dried overnight at 103-105° C  
Final weights determined  
Results calculated

Test does not require method blanks, laboratory control samples or spikes. Replicates are only required by method or client request.

Because the laboratory dries samples overnight, it only verifies the constant weight of samples once a quarter, as currently allowed. On this analysis day, the laboratory did not have to perform the constant weight verification.

**Example #5, in summary**

- 1-2. Verify Analytical Balance to gm, mg-range
- 3-4. Determine tare weights
- 5-6. Filter samples and dry
- 7-8. Determine captured weight

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## **Phosphorus Analysis Now**

Colorimetric Total Phosphorus

Calibration: 3 standards, Linear

Analytical Batch: 6 samples, digested in a single  
preparation batch

The following slides illustrate what is required  
of phosphorus analysis now.

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## **Phosphorus Analysis Now Calibration**

Initial Calibration

Standard 1

Standard 2

Standard 3

Method Blank

Samples 1-6

---

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## **Phosphorus Now Analytical Batch**

Sample 1 Replicate

Sample 1 Matrix Spike

\*\* Laboratory analyzes "Blinds" 3 times/year

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## **Do I need to change?**

### **No, if:**

1. Laboratory continues to analyze Quality Control Sample for test three times per year.
2. Matrix Spike is assessed against control limits for Laboratory Control Sample
3. Continuing Calibration Verification standard analyzed with the next batch is acceptable

A laboratory can continue to analyze phosphorus without purchasing a second source standard, and without processing an LCS, an ICV, and a closing CCV if it meets the conditions illustrated in this slide.

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## **Phosphorus Analysis Now Calibration Verification**

Colorimetric Total Phosphorus

Calibration: Verification of Calibration with "Known Standard"

Analytical Batch: 6 samples, digested in a single preparation batch

This illustrates what a laboratory does now for phosphorus analysis on a day when it does not perform a calibration.

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## **Phosphorus Analysis Now Verification Only**

Known Standard

Method Blank

Samples 1-6

Sample 1 Replicate

Sample 1 Matrix Spike

\*\* Laboratory analyzes "Blinds" 3 times/year

## Do I need to change?

### No, if:

1. Laboratory continues to analyze Quality Control Samples for test 3 times/year
2. Matrix Spike is assessed against control limits for Laboratory Control Sample
3. Continuing Calibration Verification standard analyzed with the next batch is acceptable

On a day when a calibration is not performed, a laboratory can continue to analyze phosphorus without purchasing a second source standard, and without processing an LCS and a closing CCV if it meets the conditions illustrated in this slide.

## An Even Better Option...

- Analyze Matrix Spike/Matrix Spike Duplicate to ensure results >LOQ
- Reread Continuing Calibration Verification standard (CCV) at end of analytical sequence to minimize potential for data qualification

The same CCV standard read at the beginning of the analysis run can be read as the closing CCV, assuming that the lag in time does not affect the standard's response.

## Contacts

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Feel free to contact any of us with your questions about this presentation or the proposed revision to Chapter NR 149.